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PRODUCTS DIVISION

## HIGH PRECISION FIBER DEVELOPMENT

FINAL TECHNICAL REPORT

FOR PERIOD

24 SEPTEMBER 1979 to 15 FEBRUARY 1981

CONTRACT NOO173-79-C-0341

Prepared for:

Naval Research Laboratory Washington, D. C.

Prepared by:

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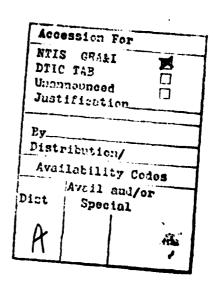
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In accordance with the statement of work (SOW) of contract NOO173-79-C-0341, EOPD has evaluated the parameters that affect the fabrication of high precision 100 um core fiber and single-mode precision fiber. <

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ITT EOPD has completed the full dimensional evaluation of precision shrunk, ground, and polished natural fused quartz tubing. Additionally, ITT has verified the od uniformity of precision shrunk, ground, and polished Vycor brand silica glass tubing. Fibers made from preforms utilizing the above substrates met all requirements of this contract. The large core deliverable had a fiber od of 140 (µm) with a fiber core diameter of 100.1 (µm,) well within the limits of the contract. Other measurements showed the core eccentricity to be less than 0.08%; optical loss at 0.85 (mm) was 7.94 dB/km while the NA was 0.29. Measurements of the low NA deliverable revealed an average NA of 0.11, an od of 80.5 (um) and a core eccentricity of 0.20%. For the high NA fiber, the NA value ran 0.20; the od was the same as the low NA, 80.5 µm, while the core eccentricity ran less than 0.20%. ( mirrome chi

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### 1.0 INTRODUCTION

The improvement of single-mode precision fiber is critical to the efficient use of these fibers in connectors and to allow for low loss splices. Similarly, the alignment of fibers to integrated optic devices demands high precision. Likewise, the precision of large core multimode fibers affects coupler and connector losses as well as overall optical and physical performance. As a result, ITT Electro-Optical Products Division (EOPD) was awarded contract N00173-79-C-0341 for the development of high prevision single-mode and large-core multimode optical fibers.

In accordance with the statement of work (SOW) of contract N00173-79-C-0341, EOPD has evaluated the parameters that affect the fabrication of high precision 100  $\mu$ m core fiber and single-mode precision fiber. This program had the following objectives:

- a. High precision multimode fiber
  - (1) Procure and evaluate precision bore, center ground, and polished WG (R) grade natural fused quartz substrate tubing for use with precision large core multimode optical fiber fabrication
  - (2) Develop techniques to fabricate precision large core, 100  $\mu m$  ±1  $\mu m$  graded index (a £10) preforms using the substrates procured during the course of task 1 efforts

- (3) Develop techniques to draw and coat the precision large core, graded index fibers that will be drawn from the high dimensional tolerance preforms fabricated during task 2
- (4) Develop the methodology necessary to evaluate and verify the fiber's high dimensional tolerances
- (5) During the contract, ship to Naval Research Laboratory (NRL) several fiber samples (100 to 200 m). These fiber samples will be used by NRL to evaluate the extent of the progress made by the contract goals. Additionally, at the end of the contract one 500 m fiber sample will be delivered to NRL
- b. High prevision single-mode fiber
  - (1) Procure and evaluate precision bore, center ground, and polished WG B grade natural fused quartz substrate tubing for axial uniformity with respect to outside diameter (od), wall thickness, bore and od ellipticity, and eccentricity
  - (2) Fabricate single-mode fiber preforms from precision ground fused quartz tubing. Measure preforms for od, ellipticity, and eccentricity
  - (3) Draw high and low numerical aperture (NA) single-mode fibers from preforms. Measure fiber od, core diameter, ellipticity, and eccentricity along fiber length
  - (4) Measure fibers for optical attenuation at 0.63  $\mu m$ , 0.85  $\mu m$ , and 1.05  $\mu m$ . Numerical aperture will be measured and fibers will be proof tested on-line during drawing

(5) Two samples of low NA fiber and two samples of high NA fiber, each 100-200 m long, will be delivered to NRL during the course of the contract. At the end of the program, one of each 0.5-1 km length of the low and high NA fiber will be delivered

All the above objectives were met. ITT EOPD has completed the full dimensional evaluation of precision shrunk, ground, and polished natural fused quartz tubing. Additionally, ITT has verified the od uniformity of precision shrunk, ground, and polished Vycor brand silica glass tubing. Fibers made from preforms utilizing the above substrates met all requirements of this contract. The large core deliverable had a fiber od of 140 µm with a fiber core diameter of 100.1 µm, well within the limits of the contract. Other measurements showed the core eccentricity to be less than 0.08%; optical loss at 0.85 µm was 7.94 dB/km while the NA was 0.29. Measurements on the low NA deliverable revealed an average NA of 0.11, an od of 80.5 µm, and a core eccentricity of 0.20%. For the high NA fiber, the NA value ran 0.20; the od was the same as the low NA, 80.5 µm, while the core eccentricity ran less than 0.20%.

Section 2.0 discusses in detail the results of the precision large core and precision single-mode fiber development efforts.

## 2.0 PRECISION FIBER DEVELOPMENT

Development of fibers having very precise dimensions was approached by examining and controlling the critical aspects of the raw materials and the fabrication process. In particular, the most important areas were determined to be substrate tube dimensions, preform fabrication conditions, and fiber draw conditions. Each of these factors is discussed in the following paragraphs.

## 2.1 Substrate Tube Dimensional Evaluation

2.1.1 Raw Fused Quartz Substrate Dimensional Evaluation
In order to obtain a prevision substrate tube having 15.00
±0.03-mm od and 1.00 ±0.02-mm wall thickness, a raw material
tube is purchased which has 16 ±0.8-mm od and 1.2 ±0.4-mm wall.
Of critical importance is the fact that the tube has a wall
thickness large enough to allow uniform grinding and polishing
after shrinking. The specified tube dimensions were selected
to meet this criterion. This raw tube is then sent to a vendor
for precision shrinking and grinding. To determine the uniformity of the raw starting material, two such tubes 38 cm long
were sliced into pieces 2 cm in length, and each piece was
measured extensively. Prior to cutting each tube, a reference
mark was scribed axially along the full tube length. In
addition, marks were scribed around the tube circumference at

2 cm intervals, and each segment was labeled with an identification number. After each tube was sliced into 19 segments with a diamond saw, the od of each segment was measured at four locations spaced 45° apart. The segment wall thickness was also measured at eight locations, spaced 450 apart. The data was recorded and is shown in Tables 2.1.1-1 and 2.1.1-2. The data is presented graphically in Figures 2.1.1-1 and 2.1.1-2 for the two tubes. The tube wall thickness and tube diameter at each measurement location are plotted as a function of axial distance along the tube. The tube in Figure 2.1.1-1 was found to have a mean wall thickness of 1.1 mm with a maximum variation of ±0.03 mm. The maximum wall thickness variation along any axial line was ±0.01 mm. By comparing the wall thickness at opposing measurement locations, the bore eccentricity was obtained. A maximum eccentricity of 0.31 was found across A-E and B-F, as calculated from 100 x (a-b)/c where a and b are opposing wall thicknesses and c is the diameter. The tube diameter was found to vary by as much as ±0.035 mm along its length, ranging from 15.94 mm at location 1 down to 15.90 mm at location 4, with a bulge of 15.97 mm occurring at location 12. The tube ellipticity was calculated as the difference between the maximum and minimum diameters divided by the mean diameter and multiplied by 100. The maximum ellipticity was found to be 0.125.

Table 2.1.1-1. Dimensional Data for Standard Substrate Tube 1.

# PRECISION FIBER DATA SHEET - SUBSTRATE EVALUATION

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TUBE PREPARATION: INSCRIBE A LINE AXIALLY ALONG TUBE--THIS IS "A" ON CROSS SECTION. INSCRIBE PIECE NUMBER ON EACH SECTION BEFORE CUTTING. Table 2.1.1-2. Dimensional Data for Standard Substrate Tube 2.

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TUBE PREPARATION: INSCRIBE A LINE AXIALLY ALONG TUBE—THIS IS "A" ON CROSS SECTION. INSCRIBE PIECE NUMBER ON EACH SECTION REFORE CUTTING.



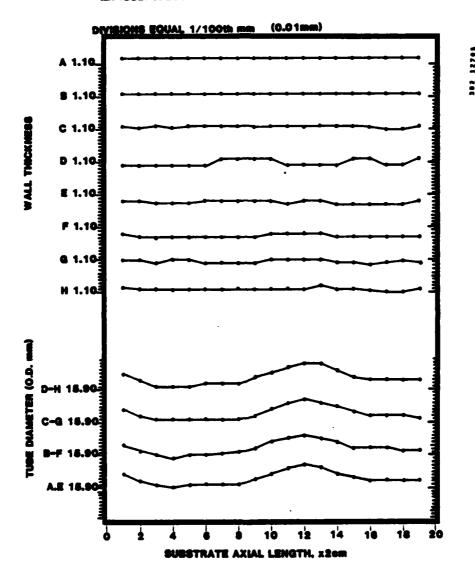


Figure 2.1.1-1. Dimensional Data Plot for Standard Substrate Tube 1.

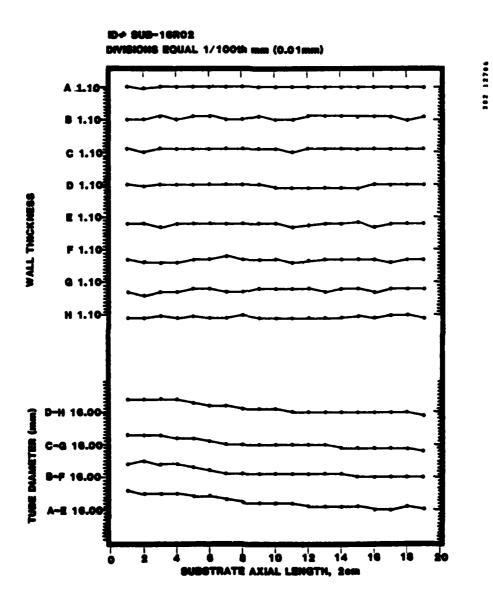


Figure 2.1.1-2. Dimensional Data Plot for Standard Substrate Tube 2.

The second raw substrate tube dimensional data, as shown in Figure 2.1.1-2, showed a mean wall thickness of 1.09 mm with a maximum variation of ±0.025 mm. The maximum wall thickness variation along any axial line was ±0.01 mm while maximum eccentricity of 0.31 was found across B-F. The tube diameter showed a gradual taper from 16.04 mm to 15.99 mm with respect to its longitudinal axis. In addition, the substrate ellipticity was found to be 0.19. The od and wall variations were thus found to be greater than desired.

- 2.1.2 Precision Fused Quartz Tube Dimensional Evaluation
  Raw substrates 91.4 cm long were sent to an outside vendor who
  shrunk the tubes onto a precision mandrel having 13.00-mm
  diameter and 76.2-cm length. The shrunk tubes were then cut
  into two 35.6 cm finished lengths and ground on centers to have
  a wall thickness of 1.0 mm. Dimensional evaluation of these
  tubes was performed for two principal reasons:
  - a. To determine od and wall thickness uniformity in order to compare preform and fiber dimensional variations to variations in the starting substrate
  - b. To ascertain the reliability of measurements made on tube ends in predicting wall thickness of the tube in the middle portion

Two of these tubes were marked, cut, and evaluated as described in paragraph 2.1.1. The raw data listed in Tables 2.1.2-1 and 2.1.2-2 are also presented graphically in Figures 2.1.2-1 and 2.1.2-2. The uniformity of these tubes is readily apparent. The mean wall thickness of the tube in Figure 2.1.2-2 was 0.98 mm with maximum variation of ±0.02 mm, while the maximum variation along any axial line was ±0.015 mm. The bore eccentricity was found to be 0.13. The tube diameter varied by a maximum of ±0.01 mm along any axial line while the maximum tube ellipticity was 0.07. Although this tube was quite uniform in od and wall thickness, the od was not within the goal of 15.00 ±0.02 mm, especially over the region from section 1 through 8. More importantly, the data seems to suggest some tube end taper in the first measured segment at locations A-E and at H and in the od at section 18.

The second precision tube evaluation confirmed the presence of a tube-end taper which is seen in the od measurement at locations 1 and 18. This tube did meet the specification of 15.00 ±0.02-mm od and 1.00 ±0.02-mm wall and exhibited excellent eccentricity and ellipticity values of 0.13 and 0.07, respectively. These results indicate that the precision tubing dimensions along the full tube length may be accurately predicted from end

Dimensional Data for Precision Substrate Tube 1. Table 2.1.2-1.

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SUBSTRATE EVALUATION
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	•	0.975	0.985	0.985	0.985	0.99	0.995	0.99	0.99	0.985	0.965	0.99	D. 99	0.99	0.99	D.99	0.985	8.	8.	
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	1	0.975	0.975	0.97	975	0.975	0.98	0.975	96.0	0.975	76.0	0.975	26.0	0.97	76.0	0.97	0.975	0.975	0.97	
	Ø	9.8	96.0	0.967	0.985	0.985	96.0	0.98	0.985	0.99	96.0	0.98	98.0	6.0	96.0	D.975	96.0	96.0	0.965	
	I	0.98	0.99	0.985	0.985	0.985	0.985	0.99	0.965	0.985	0.985	0.985	0.985	0.985	96.0	0.985	0.98	0.98	96.0	
8	A-E	7.8	14.96	14.96	14.96	14.96	14.96	14.96914	14.97	14.97	14.97	14.97	14.97	14.97	14.97	14.97	14.97	14.97	4.97	
_	8-F	1.96	34.96	14.96	4.965	14.97	14.97	4.8	14.97	14.97	14.97	14.98	14.98	14.97	14.97	4.975	14.97	8.	4.97	
	9-0	14.96	14.97	14.96	14.97	14.97	14.97	14.97	14.97	14.97	14.98	14.98	14.98	14.98	8.4	14.98	14.98	4.98	14.97	
	H	D-H 14.96	8.	24.98	2.9	7.96	14.96514.	14.965	1.965	14.97	14.97	14.97	14.97	14.97	14.97	14.97	14.975.4.97	14.97	8.	
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		•	S&P-15E03	5603	=	10														

TUBE PREPARATION: INSCRIBE A LINE AXIALLY ALONG TUBE--THIS IS "A" ON CROSS SECTION. MSCRIBE PIECE NUMBER ON EACH SECTION BEFORE CUTTING. Dimensional Data for Precision Substrate Tube 2. Table 2.1.2-2.

# PRECISION FIBER DATA SHEET - SUBSTRATE EVALUATION

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	m	0.995	0.995	96.0	0.99	0.99	0.995	0.99	0.995	0.99	.93 2.93	0.99	8.6	0.995	0.995	3.995	0.995	0.996	8.	
	4	0.985	96.q	D. 985	9.6	0.985	0.99	0.985	0.985	9. O	96.0	96.0	96.0	0.99	96.0	88.	0.965	98.0	8.0	
	O	0.995	96.0	96.0	3.985	0.985	0.985	0.99	0.985	0.985	88.0	98.0	98.0	0.995	88.	88.	0.985	98.	98.0	
	Ξ	8.	D. 995	00.	3.995	0.995	0.995	0.99	0.99	0.99	0.99	0.99	0.99	0.99	96.0	93	0.9	0.995	0.995	
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<	¥-4	2.8	15.03	14:99	2.00	5.8	_	15.0015.005	15.00	15.00	15.00	15.00	4.99	15.00	5.00	5.00	14.995	15.00	4.99	
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TUBE PREPARATION: INSCRIBE A LINE AXIALLY ALONG TUBE-THIS IS "A" ON CROSS SECTION. INSCRIBE PIECE NUMBER ON EACH SECTION BEFORE CUTTING.



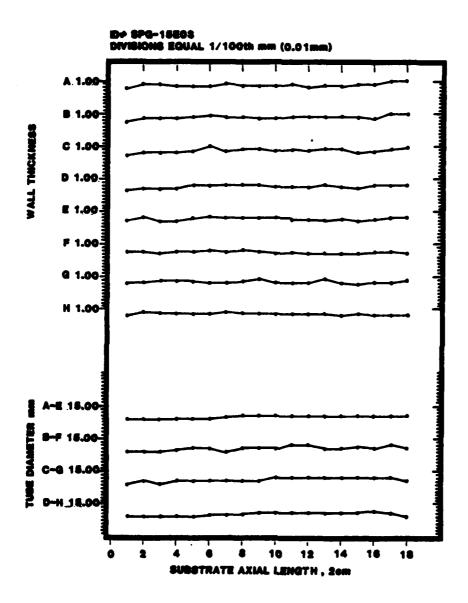


Figure 2.1.2-1. Dimensional Data Plot for Precision Substrate Tube 1.

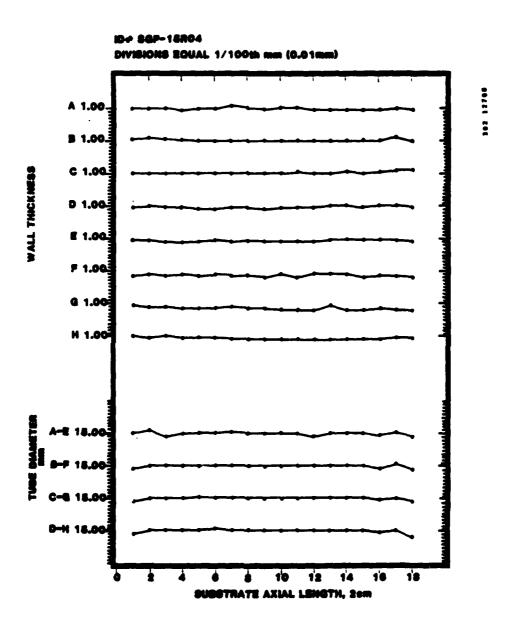


Figure 2.1.2-2. Dimensional Data Plot for Precision Substrate Tube 2.

measurements if those measurements are taken at a distance  $\geq 2$  cm from the tube ends. The results also indicate that a classification and selection process should be used for tubes utilized in precision fiber development, so that equal cross-sectional areas for tubes are ensured. This is necessary to achieve reproducible core-to-od ratios from preform to preform.

# 2.1.3 Borosilicate Tubing

An important end use for precision large core fibers is in the fabrication of low loss couplers. Since the present coupler design requires the use of fibers having Vycor cladding, a request was submitted to NRL and approved to allow the use of precision Vycor as substrate for the large core fibers instead of the precision natural fused quartz. The nominal size of the raw Vycor (type 7913) tubes as purchased was 19-mm od with a 1.2-mm wall. Tubes of this type were precision shrunk and ground to 15-mm od and a 1.0-mm wall. Since previously evaluated quartz tubes met the dimensional goals and since the Vycor tubes were shrunk onto the same mandrels, it was concluded that only a check of od uniformity was necessary to qualify the prevision Vycor tubes. Three precision Vycor tubes were measured at 2-cm intervals along their length for od ellipticity and axial

od uniformity. The tubes were within specifications and were as good dimensionally as the previously measured quartz tubes. Table 2.1.3-1 summarizes the dimensional data for these tubes.

2.2 Preform Design Fabrication and Dimensional Analysis
There were three separate fiber types studied on this program.
Individual paragraphs below discuss aspects of preform design
and fabrication followed by dimensional analysis for each preform
type.

## 2.2.1 Large Core Multimode

Intended for short haul data bus or interconnect applications, the large core preforms were designed to provide high coupling efficiency while exhibiting moderate bandwidths and attenuation properties. To meet these design goals, a 100  $\mu m$  core/140  $\mu m$  od fiber was selected with a minimum NA goal of 0.25. In addition, the profile alpha parameter of 10 was selected. This preform used prevision WG  $^{\textcircled{R}}$  substrate and had a depressed borosilicate optical cladding layer and a germania and phosphorous-doped core.

Later in the program the preform design was changed to facilitate usage of the fibers in a biconical fused taper coupler. At this time, a precision borosilicate tube was used both as a support

Table 2.1.3-1. Borosilicate Tubing Dimensional Data.

# Tube Diameter (mm)

	Tub	<u>e 1</u>	Tub	<u>e 2</u>	Tub	<u>e 3</u>
Lctn	Max	Min	Max	Min	Max	Min
1	15.06	14.96	14.96	14.96	14.98	14.96
2	14.96	14.94	14.96	14.96	14.98	14.96
3	14.96	14.96	14.98	14.96	14.98	14.96
4	15.00	14.96	14.98	14.96	15.04	14.98
5	14.98	14.98	15.00	14.98	15.00	14.98
6	14.96	14.96	15.02	15.00	15.00	14.98
7	14.96	14.96	15.00	15.00	15.00	14.96
8	14.96	14.96	14.98	14.98	14.98	14.98

tube and for the optical cladding. That is, the core material was deposited directly into the tube without an intermediate optical cladding which had been shown to be a barrier to efficient coupling. In order to provide a smooth core index profile, the core deposited next to the substrate consisted of germanium, phosphorous, and boron oxides in a mixture which index matched the substrate tube. Then the germania content was increased and the boron content was decreased toward the core center, both to fit an alpha equal to 5 profile. This lower alpha value was effective in reducing the pulse dispersion below the 10 ns/km goal while still providing enough step index character to permit efficient coupling.

The fabrication effort on the large fiber preforms was directed toward adjustment of chemical flows and pass numbers to achieve the specified NA and core diameter values. Early in the program, large core fiber preforms were fabricated using precision shrunk and ground WG substrate tubes and using optical pyrometer feedback temperature control. The initial results revealed that the core-to-od ratio was approximately 7% below the desired value of 0.71. The cane NA values were approximately 10% below the minimum NA required by the contract. To improve the fiber properties which were to materialize from the preforms, new preform

designs were made wherein the parameters were the NA and the core diameter. Before this fabrication process could be fully characterized, the design approach was changed as described above and in the following paragraph.

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Later in the program, the substrate tubing was switched from WG  $^{\circledR}$  to Vycor  $^{\circledR}$  to facilitate coupler assembly. Twenty-three preform starts were made during this time frame. Of this number, five shattered at the start of collapse. Over 50% of the remainder had low yields because of bubbles in the preform. At the beginning of the fabrication with the Vycor  $^{\circledR}$  the core diameter usually averaged 96  $\mu$ m. At the end of the program enough changes had been made in the preform fabrication to bring the average up to 99  $\mu$ m. The NA has constantly run between 0.29 and 0.30.

A great deal of the fabrication effort was spent on problems associated with the preform bubbles. The major sources of these bubbles were found to be associated with the substrate tube. Typically, in bad tubes, small bubbles or seeds appeared as specks before deposition began. During collapse, these bubbles would grow very large and burst through the outside of the preform, distorting both the core and the preform surface. The bad tubes were found to run in batches wherein some cartons of tubes

tended to have a large number of defective tubes while other cartons had more good tubes. It was found that yields could be improved somewhat by illuminating the tubes with a high intensity light source and eliminating tubes having a high concentration of defects.

## 2.2.2 Low NA Single-Mode Fiber

The preform design goals for this fiber included a material NA of 0.10, a core diameter of 4.4  $\mu m$ , and an od of 80  $\mu m$ . The NA and core diameter were measured on a multimode cane pulled from the completed preform. These parameters were obtained by depositing a borosilicate optical cladding of about 30  $\mu m$  thickness (in fiber) followed by a step index germanosilicate core. Adjustments to the NA were made by varying the borosilicate concentration.

The preform fabrication proceeded by reducing the NA from 0.112-0.119 down to 0.10, as shown in Table 2.2.2-1, followed by adjustment of the deposition passes to give a core diameter of 4.4  $\mu m$  in an 80  $\mu m$  fiber.

Table 2.2.2-1. Normalized Preform Dimensions: Low NA Single-Mode Fibers.

Preform No	NA	Core Dia (µm)	Fiber Dia (um)
21127	0.12	3.4	80
21153	0.12	3.5	80
21174	0.12	4.1	80
21178	0.11	3.9	80
21182	0.11	3.8	80
21186	0.10	4.0	80
21219	0.10	4.0	80
21227	0.11	4.5	80
21229	0.12	3.8	80
21236	0.10	4.4	80
21247	0.11	4.5	80

# 2.2.3 High NA Single-Mode

The design goals for this fiber were material NA of 0.20, core diameter of 2.2  $\mu m$ , and an od of 80  $\mu m$ . The NA and core were as measured on multimode preform canes. The design approach in comparison to the low NA fiber included high concentration of boron and germania in the cladding and core, respectively.

As fabrication proceeded, the NA values were increased from about 0.18 to the desired value of 0.20, as shown in Table 2.2.3-1, and the core size was adjusted to 2.2  $\mu m$  in 80  $\mu m$  by varying both the number of deposition passes and the chemical flow rates.

### 2.3 Fiber Fabrication and Evaluation

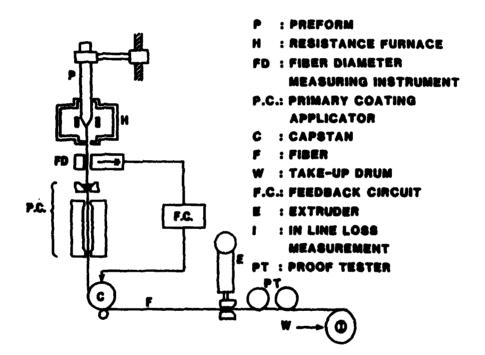
After the prevision preforms were fabricated, they were drawn into fiber as discussed below. The fibers were then evaluated for dimensional stability as well as for conformance to the performance goals. In addition, the optical attenuation of the fibers was measured.

## 2.3.1 Fiber Drawing

All precision fibers were drawn on a rig as diagrammed in Figure 2.3.1-1 using a furnace with a graphite resistance heating element as the draw heat source. The fiber od was continuously

Table 2.2.3-1. Normalized Preform Dimensions: High NA Single-Mode Fibers.

Preform No	MA	Core Dia (µm)	Piber Dia (µm)
21165	0.10	1.9	80
21167	0.11	2.9	80
21176	0.13	2.4	80
21180	0.19	1.7	80
21183	0.19	2.2	80
21187	0.19	2.2	80 .
21228	0.17	2,5	80
21231	0.20	1.9	80
21241	0.20	2.3	80
21257	0.20	2.2	80



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Figure 2.3.1-1. Optical Fiber Drawing Apparatus.

monitored with a laser micrometer at a measuring rate of 1000 scans per second with a precision of  $\pm 0.3~\mu m$ . The fiber od was controlled with a closed loop feedback system which controlled the pulling speed and was linked to the laser micrometer. A strip chart recorder was used to provide a printed record of the fiber diameter about the preselected nominal od and, in general, was used to calculate the overall diameter control in the form of plus or minus percent deviation from the nominal.

Fibers were primary coated with a thermally cured RTV silicone resin to a nominal diameter of 200 µm and secondary coated with Hytrel polyester to a nominal diameter of 400 µm. Early in the program, the accuracy of the diameter strip chart was verified by cutting fiber sections at various regular intervals for fiber od measurement using a mechanical micrometer. Measurement intervals of 1 m, 10 cm, and 1 cm were employed for this purpose. Table 2.3.1-1 is a partial summary of the results of these measurements indicating fiber diameter control. It should be noted that the plus or minus percent figure is derived from the quotient of the standard deviations and the mean of the measured values and does not represent either the maximum deviation or the standard deviation from the nominal od. The percent maximum deviation is generally somewhat greater than the percent standard

Table 2.3.1-1. Piber Diameter Measurements.

			=	1 m Intervals	118	10 CM	10 cm Intervals	vals	1 6	1 cm Intervals	•t
	Piber No	Nominal od	Nean (vm)	Std Devn (um)	## Std Devn	Mean (um)	Std Devn (##)	std Devn	Mean (FM)	Std Devn (#M)	std Bevn
	790727-3	127	126.0	1.13	06.0	125.4	125.4 1.14 0.91	0.91	•	1	ı
	790726-2B	127	ı	ı	ı	126.2	1.02	0.81	1	ı	ı
	791217-5	127	127.6	0.98	0.76	ı	1	ı	128.23	0.941	0.73
	791220-2	127	127.2	0.475	0.37	t	1	1	127.25	0.65	0.51
	791227-3	140	ı	ı	ı	1	ı	•	140.3	1.09	0.78
27	800110-5	78.7	79.0	0.03	0.89	ŧ	1		78.7	0	0

deviation. For instance, for fiber 791227-3 in Table 2.3.1-1 at 1-cm intervals, the percent standard deviation is  $\pm 0.78$  and the percent maximum deviation is  $\pm 1.38$ .

Figures 2.3.1-2 and 2.3.1-3 are strip chart records of the diameter control for two fibers. Figure 2.3.1-2 shows the diameter record for a 140-µm nominal od fiber with the diameter control as indicated. The numbers on the charts are mils (0.001 in) and the charts were recorded at 12 in/h. Figure 2.3.1-3 is the record of a 75-µm nominal od single-mode fiber with diameter control as indicated. Neither of the preforms from which these two fibers were drawn was fabricated from precision tubing.

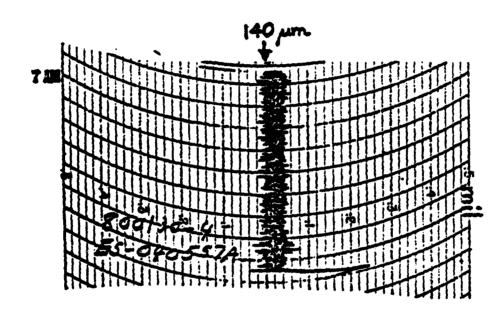


Figure 2.3.1-2. Diameter Monitor Chart for 140-um od Fiber.

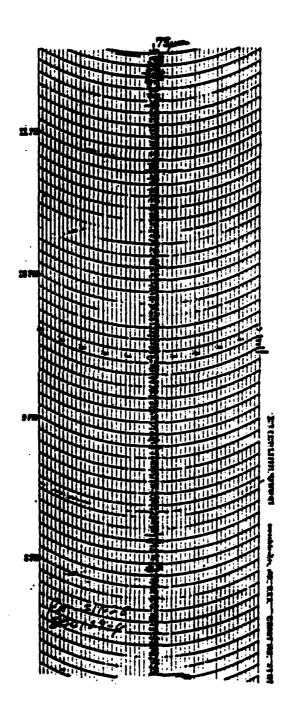


Figure 2.3.1-3. Diameter Monitor Chart for Single-Mode Fiber.

## 2.3.2 Fiber Dimensional Evaluation

During this period, techniques were investigated for evaluation of core and outside diameters as well as core and od ellipticity and eccentricity. An optical microscope with a vernier eyepiece was planned for the large core evaluation while scanning electron microscopy (SEM) photographs were planned for the single-mode fiber measurements.

The transmission optical microscope technique was attempted on two occasions. Each time the measurements were unsatisfactory because of fiber drift due to relaxation of the fiber holding mechanism and also because of drift of the microscope stage. At that point it was decided to utilize SEM for the large core fibers as well as the single-mode fibers.

Fiber ends were prepared for SEM photographs by scribing the fiber lightly with a diamond and breaking the fiber under axial tension. This was followed by hydrofluoric acid etch for 5 to 10 s. Both large core and single-mode fiber ends were photographed using SEM and the images were measured along their horizontal (x) and vertical (y) axes with vernier calipers. Five successive measurements were made of each dimension of the

images to establish the measurement uncertainty, which is reported as the mean of the variances of each measurement from the mean and the mean percentage variance. The reference length printed on each photomicrograph was also successively measured and the uncertainty calculated. Finally, the actual dimensions were calculated along with their total unc rtainties. This data is summarized in Table 2.3.2-1. Two significant items are noted. First, all dimensions can be evaluated with a precision of better than 1%, except for the core measurement of the single-mode fiber. Second, the accuracy of the SEM was questioned because of the large fiber ovality indicated since optical microscope photographs had always indicated very good fiber circularity. Fiber draw data was inspected for the two fibers discussed and values of 75  $\mu m$  for the single-mode fiber and 140  $\mu m$  for the large core fiber were found. It appears that the SEM photomicrographs may be dimensionally accurate along the vertical (y) axis but are compressed along the horizontal axis. At this time, the SEM equipment manufacturer was contacted to repair the device. The factory representative calibrated the SEM and found it to be accurate only on the x-axis and within 2%. He also found that the accuracy changes with but is not a function of the magnification factor.

Table 2.3.2-1. Piber Dimensional Analysis.

Fiber	Dimension		Photomic: X-Axis	Photomicrograph Image Measurement (mm)	e Measur	ement (mm) Y-Axis	
Type	Measured	Mean	Mean Variance	Percent	Mean	Mean Variance	Percent
rc*	8	81.028	0.038	0.046	86.552	0.082	0.094
	core	50.604	0.117	0.231	54.028	0.070	0.129
	reference (50 µm)	30.632	0.042	0.138	1	1	•
** MS	po	79.902	0.034	0.042	85.938	0.034	0.007
	core	2.992	0.026	0.856	3.122	0.040	0.231
	reference (5 µm)	5.980	0.028	0.468	ı	i	1

Dimension Calculated Using Photo Reference (µm)

X-Axis	Percent Uncertainty	28 0.23	19 0.26	185 0.51	2.61 0.70	
	nt inty Diameter	141.28	88.19	71.85		
X-Axis	Percent er Uncertainty	6 0.18	0 0.37	1 0.51	0 1.32	
	Diame	132.26	82.60	66.81	2.50	
	Fiber Dimension		core	Po **WS	Mode core	
ĺ		3		S	N.	

\*Large core.

Fiber sample mounting techniques were investigated so that the ellipticity and eccentricity of fibers could be measured. With the goal for fiber diameter being 80  $\pm 0.5~\mu m$  or  $\pm 0.68$ , the 28 accuracy limit continued to provide uncertainty about the actual fiber diameter and other dimensions.

At this point, a number of techniques were compared for their reproducibility as discussed below. A summary of this dimensional precision study appears in Table 2.3.2-2.

One fiber od measurement technique was developed which used the laser micrometer on the draw tower. Fiber sections are placed in a fixture to hold them rigidly in the laser beam. Measurement variation for the laser micrometer is reported to be no greater than 0.22  $\mu$ m over a wide variety of orientations. The accuracy is checked using standards supplied by the vendor.

Other dimensional evaluation techniques used on this program included the use of a digital micrometer. After several fiber samples were checked, the main problem that became evident was the lack of reproducibility. Of all the methods evaluated this one had the highest standard deviation. The reason this

Table 2.3.2-2. Comparison of Dimensional Evaluation Methods.

<u>Method</u>	Mean	Standard Deviation
Digital micrometer	78.2 µm	1.09
Anritsu	80.95 µm	0.07
Measurements laboratory	81.9 µm	0.28
Laboratory micrographs	81.8 µm	-
Scanning Electron Microscope	76.0 µm	-

procedure gave the poorest results is that it involves more human judgment as to how or where the fiber sample was placed in the micrometer and also how much pressure was applied to the sample.

The final technique was utilized by the ITT measurements laboratory. This method involves taking a photograph of the end of the fiber and using calipers to measure the core and outside diameters of the samples. One of the biggest sources of error with this method is judging where the core ends and the cladding begins on the sample. Another error source is the likelihood that the technician will erroneously read the calipers. These conclusions were verified when this procedure had the second highest standard deviation of all the techniques evaluated.

Since the laser micrometer gave the most consistent results, considerable effort was spent using this method to measure od on both long and short length fibers. For example, on low NA single-mode preform 21229, a 500-m section was measured every 10 m (see Figure 2.3.2-1). The average od was 80.5  $\mu$ m with a maximum variation of 2.0  $\mu$ m and a nominal variation of ±0.36  $\mu$ m. The 2.0- $\mu$ m variation was due to two points in the total length of the

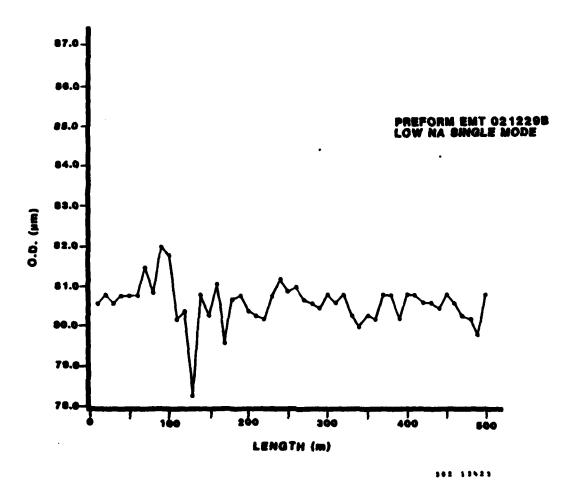


Figure 2.3.2-1. Diameter of Low NA Fiber Each 10 m.

fiber and is not a regular occurrence. When a 10-m sample of the same fiber was checked every meter (see Figures 2.3.2-2 and 2.3.2-3), the average od was 80.5  $\mu$ m, the maximum variation from the average was 0.6  $\mu$ m, and the nominal variation was  $\pm 0.25 \ \mu$ m. When a 1-m sample was checked every 10 cm (see Figures 2.3.2-4 and 2.3.2-5), average od was 80.7  $\mu$ m, with a maximum deviation from the average of 0.4  $\mu$ m and a nominal variation of  $\pm 0.14 \ \mu$ m. For a 10-cm section checked every centimeter (see Figures 2.3.2-6 and 2.3.2-7), the average od was found to be 80.7  $\mu$ m, a maximum variation of 0.2  $\mu$ m, and a nominal variation of  $\pm 0.2 \ \mu$ m.

On high NA single-mode preform 21228, a 10-m section was measured every meter (see Figure 2.3.2-8). The average od was 80.5  $\mu$ m with a maximum variation of 0.6  $\mu$ m and a nominal variation of  $\pm$ 0.26  $\mu$ m. When a 1-m sample was sampled every 10 cm (see Figure 2.3.2-9), the average od was found to be 80.9  $\mu$ m with a maximum variation of 0.2  $\mu$ m and a nominal variation of  $\pm$ 0.05  $\mu$ m. For a 10-cm section sampled each centimeter (see Figure 2.3.2-10), the average od was 80.8  $\mu$ m with a maximum variation from the average of 0.3  $\mu$ m and a nominal variation of  $\pm$ 0.1  $\mu$ m.

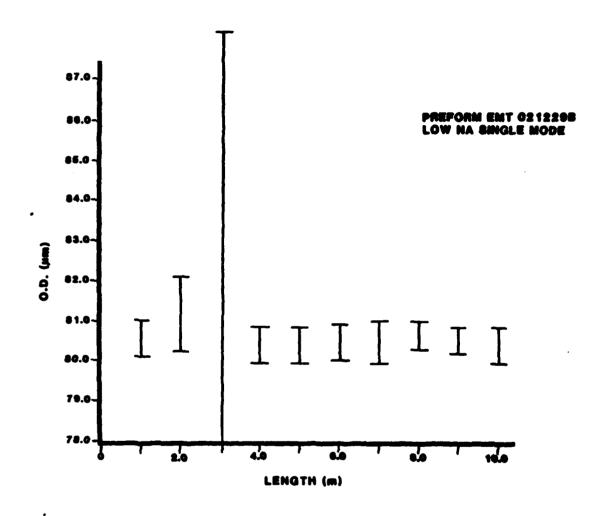
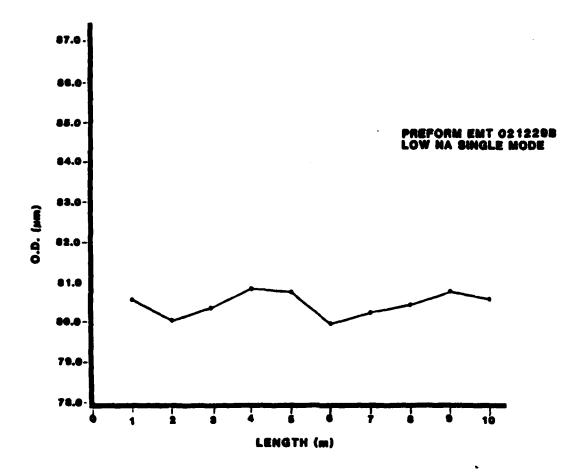


Figure 2.3.2-2. Diameter of Low NA Fiber Each Meter.



Pigure 2.3.2-3. Mean Diameter of Low NA Fiber Each Meter.

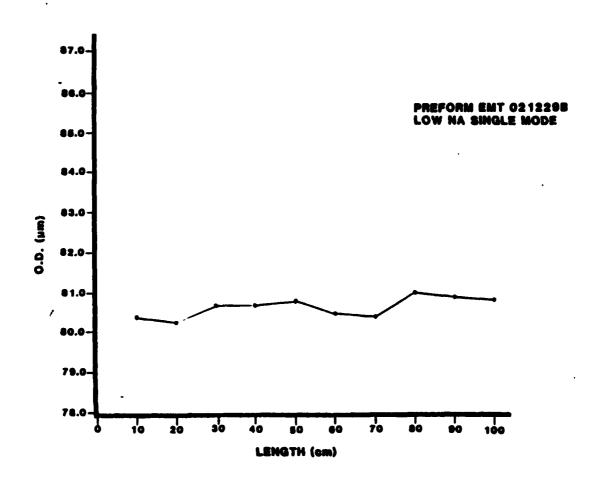


Figure 2.3.2-4. Mean Diameter of Low NA Fiber Every 10 cm.

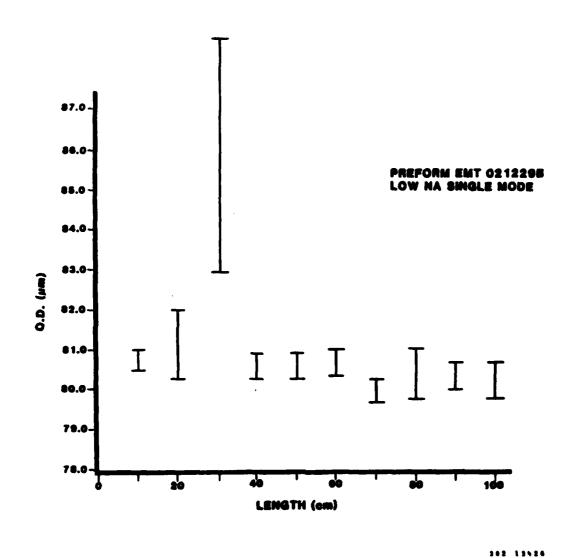


Figure 2.3.2-5. Diameter of Low NA Fiber Every 10 cm.

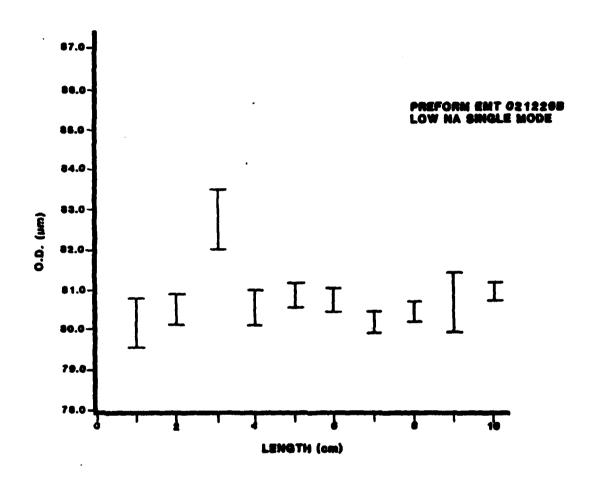


Figure 2.3.2-6. Diameter of Low NA Fiber Every Centimeter.

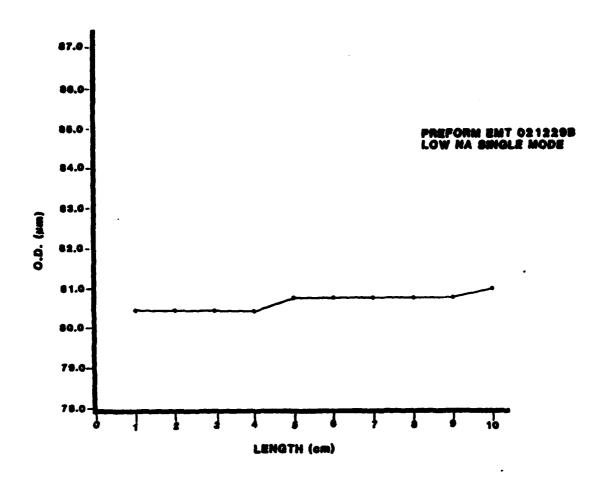


Figure 2.3.2-7. Mean Diameter of Low NA Fiber Every Centimeter.

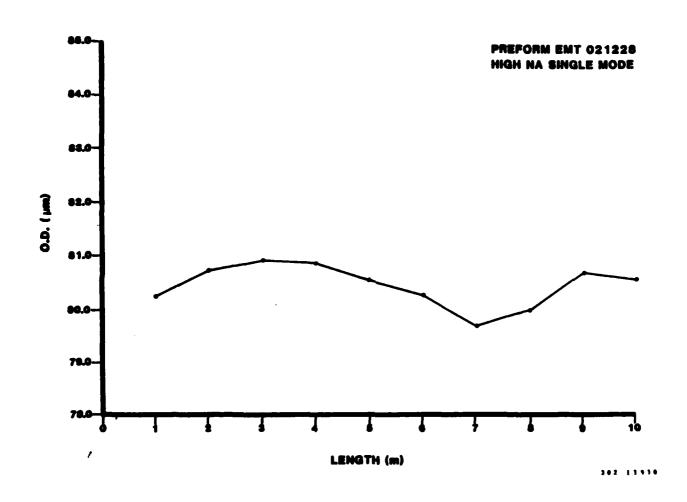


Figure 2.3.2-8. Mean Diameter of High NA Fiber Every Meter.

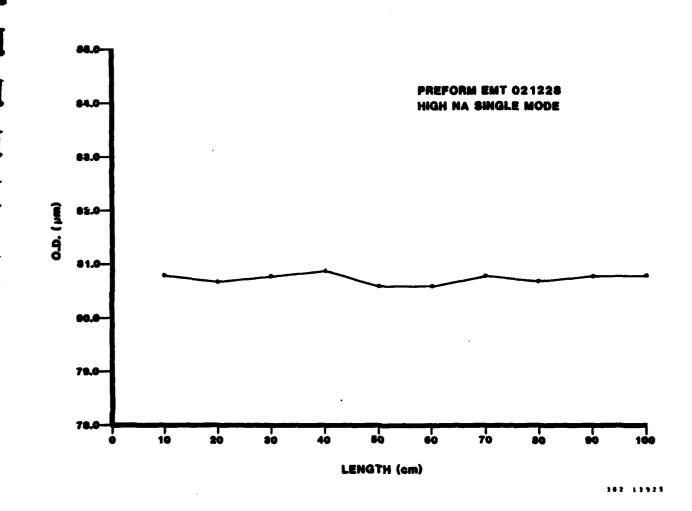


Figure 2.3.2-9. Mean Diameter of High NA Fiber Every 10 cm.

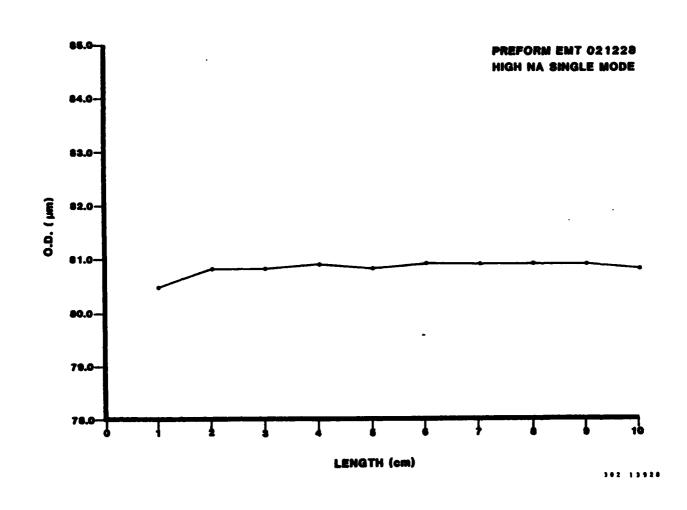


Figure 2.3.2-10. Mean Diameter of High NA Fiber Every Centimeter.

When the od of large core preform 21235 was evaluated in a similar manner, the following results were obtained for a 500 m fiber sampled every 10 m (see Figure 2.3.2-11); the section had an average od of 140.2  $\mu$ m, a maximum variation from the average of 1.3  $\mu$ m, and a nominal variation of  $\pm 0.35$   $\mu$ m. When a 10-m section was sampled every meter (see Figure 2.3.2-12), the average od was 140.5  $\mu$ m with a maximum variation of 0.8  $\mu$ m, and a nominal variation of  $\pm 0.32$   $\mu$ m. A 1-m sample every 10 cm (see Figure 2.3.2-13), had an average od of 140.5  $\mu$ m with a maximum variation of 0.2  $\mu$ m and a nominal variation of  $\pm 0.1$   $\mu$ m. When a 10-cm section was checked every centimeter (see Figure 2.3.2-14), the average od was 140.3  $\mu$ m with a maximum variation from the average of 0.3  $\mu$ m and a nominal variation of  $\pm 0.16$   $\mu$ m.

Evaluation of absolute core diameter in the single-mode fibers proved to be an extremely difficult task. When near-field photomicrographs were viewed, a diffuse core-clad interface was seen which could not be used to determine a precise core dimension. Likewise, the SEM was not felt to be accurate for this application. However, since axial dimensional uniformity was a major goal of this program, the SEM was used to measure the core precisely in both high and low NA single-mode fibers.

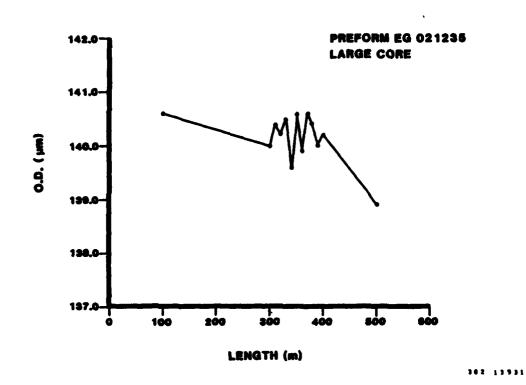


Figure 2.3.2-11. Mean Diameter of Large Core Fiber Over 500 m.

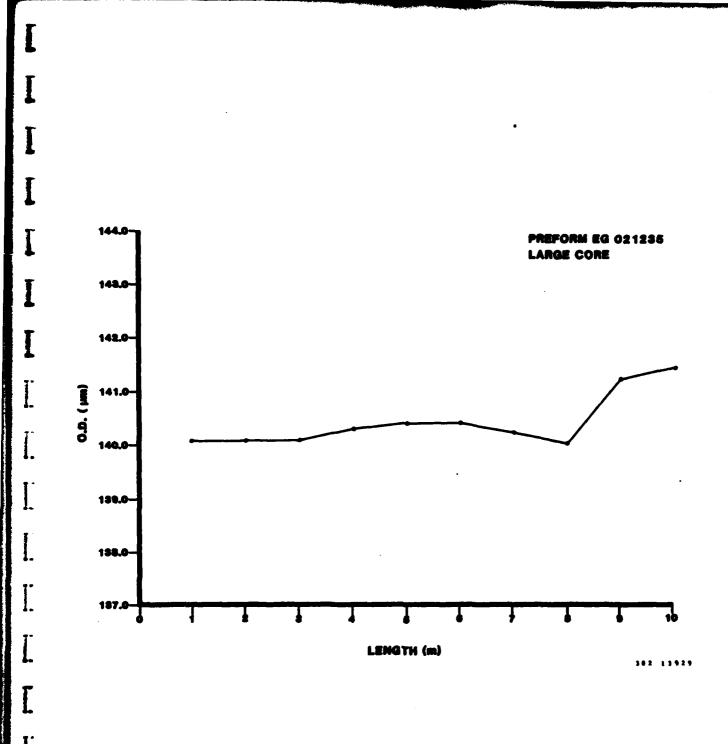


Figure 2.3.2-12. Mean Diameter of Large Core Fiber Every Meter.

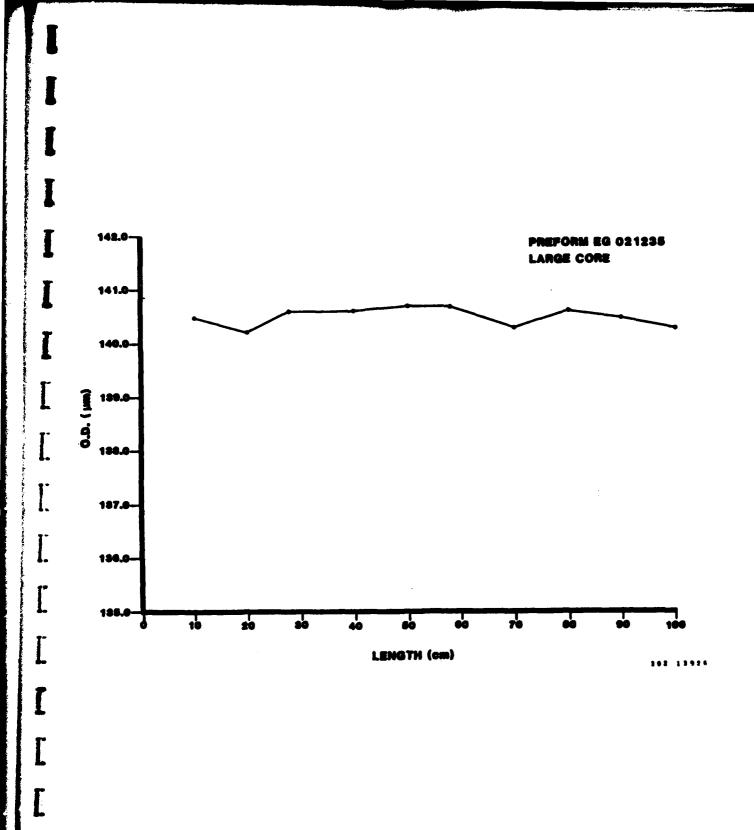


Figure 2.3.2-13. Mean Diameter of Large Core Fiber Every 10 cm.

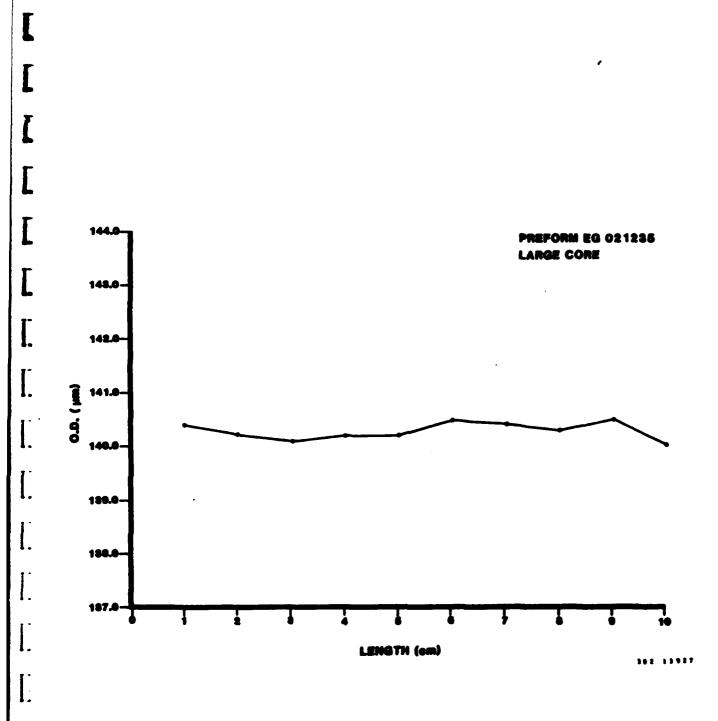


Figure 2.3.2-14. Mean Diameter of Large Core Fiber Every Centimeter.

A series of large images produced by the SEM (as shown in Figure 2.3.2-15) for an acid etched low NA fiber was measured over a 500-m fiber length.

The core diameter as measured from these photomicrographs was found to be 5.3  $\mu m$  on average with a standard deviation of 0.07  $\mu m$  and a maximum variation of 0.1  $\mu m$  as illustrated in Figure 2.3.2-16. This core diameter should not be construed as an absolute value since the selection of the core-clad interface was an arbitrary but reproducible one (which may or may not correspond to the optical core boundary).

For reference, a similar photomicrograph of an acid-etched high NA fiber is shown in Figure 2.3.2-17. It should be noted that the core is smaller as expected and that a greater degree of etching occurred in the more highly doped cladding region.

To summarize the single-mode fiber core diameter evaluation, it appears that an accurate measurement using physical techniques may not be practical or even necessary. In retrospect, the best fiber characterization may include an NA measurement along with a second mode cut-off determination wherein the core

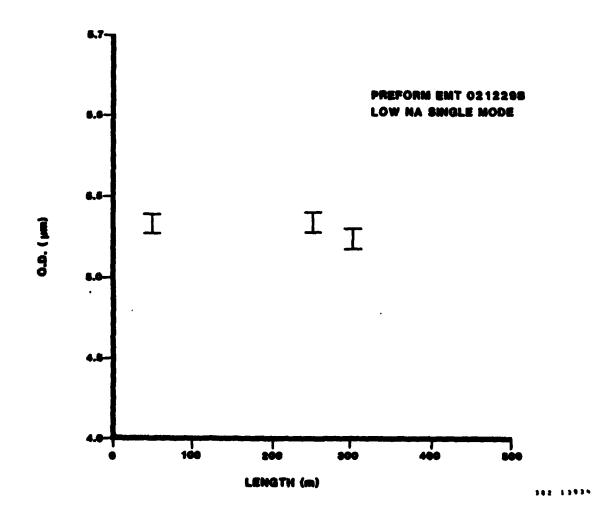


Figure 2.3.2-15. Core Diameter Versus Length for Low NA Single-Mode Fiber.

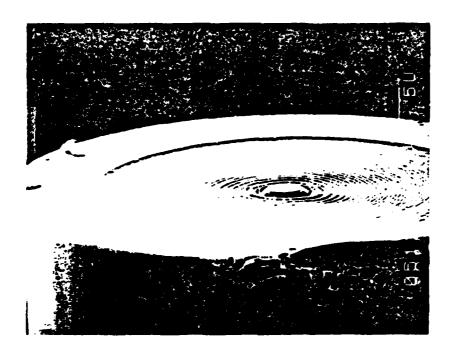


Figure 2.3.2-16. SEM Photomicrograph of an Etched Low NA Single-Mode Fiber.

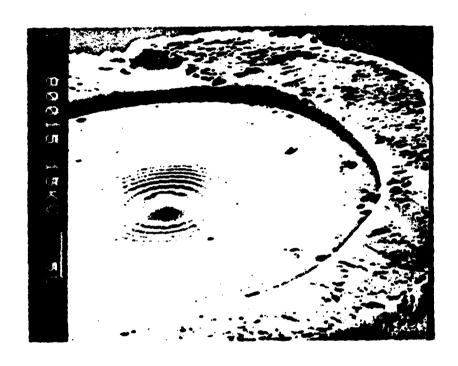


Figure 2.3.2-17. SEM Photomicrograph of an Etched High NA Single-Mode Fiber.

diameter becomes a general value describing a dimensional range. Although second mode cut-off determinations were not planned for this program, some were done as will be described in paragraph 2.3.3.

Single-mode fiber eccentricity was also evaluated from end-on SEM photomicrographs of an etched low NA fiber. The data, which is shown in Figure 2.3.2-18, indicates that the use of precision tubes is effective in producing fibers with very little eccentricity over 500 m (within the contract goal of 0.25%).

Evaluation of the large core fiber core diameter was also difficult to perform again because of the diffuse core-clad interface. The methods of near-field photomicrograph measurement and refractive-index profile were tested and compared. A refractive-index profile of fiber EG-21394 appears in Figure 2.3.2-19. Even though the selection of the point at which the core-clad interface occurs is arbitrary, this method does give distinct boundaries which can be precisely measured.

For example, the refractive-index profile of preform EG21394 was used to determine core size in the following manner. Using

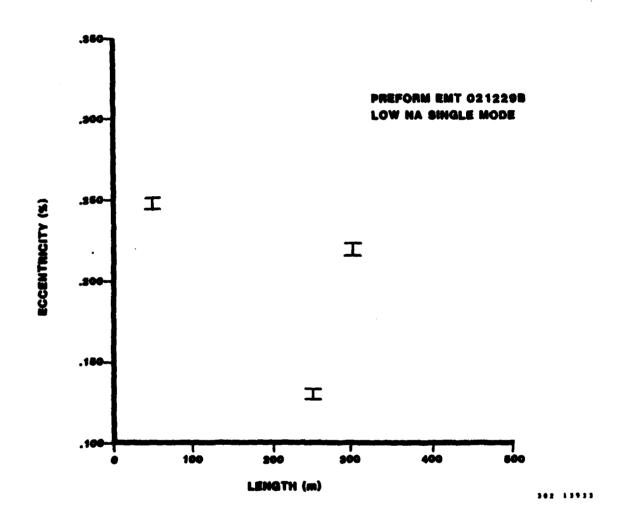


Figure 2.3.2-18. Single-Mode Fiber Core.

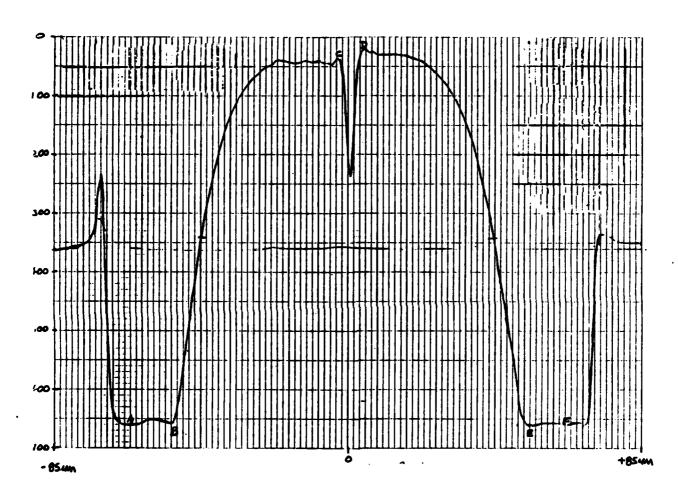


Figure 2.3.2-19. Refractive Index Profile of Large Core Fiber.

a pair of calipers the ratio of core diameter to fiber od was derived. The result was then multiplied by 170  $\mu m$ . For this particular fiber the core size was determined to be 100.1  $\mu m$ . This value compares with 98.1  $\mu m$  derived from an ITT measurements laboratory photograph.

## 2.3.3 Fiber Optical Evaluation

All of the fibers fabricated during this program were evaluated for attenuation and NA and in addition the large core fibers were evaluated for pulse dispersion. This data is discussed below.

Table 2.3.3-1 lists fiber data for the large core multimode fibers. The NA is shown to be tightly controlled at 0.29 to 0.30 as desired. The attenuation was typically less than the 10 dB/km goal, and the pulse dispersion was also typically less than the 10 ns/km goal. Dispersion was not measured in short fibers. Most of these fibers were shipped as residuals to the Interface Technology Group at ITT EOPD for use in the Navy's Coupler MTP program.

Table 2.3.3-1. Precision Large Core Multimode Fiber Data.

Preform No	Length (m)	Core Dia	<u>na</u>	Loss at 0.85 µm (dB/km)	Dispersion at 0.91 µm (ns/km)
EG 21413	745	99 x 97	0.14	Not Tested	-
EG 21432	203	96 x 100	-	7.82	-
EG 21300	597(4)*	84 x 83	0.30	4.77	-
EG 21403	394(4)*	111 x 106	0.29	5.65	-
EG 21420	730	97 x 91	0.29	10.39	6.83
EG 21319	377(3)	96	0.29	7.99	-
EG 21380	100	101 x 99	0.28	12.90	-
EG 21316	1000(2)	91 x 94	0.29	5.62	11.21
EG 21328	775	97	0.30	6.83	8.92
EG 21343	580(2)	100	0.29	11.50	-
EG 21341	560(3)	100 x 101	0.29	7.60	-
EG 21407	50	91 x 98	0.29	NE**	-
EG 21362	50(2)	97 x 96	-	NE**	•
EG 21397	105	99 x 97	0.30	NE**	~
EG 21394	968	99 x 98	0.29	7.94	8.92
EG 21467	130	95	0.30	14.12	-
EG 21498	670(2)	94 x 103	0.30	10.72	-
EG 21508	630	101 x 102	0.29	3.79	5.44

<sup>\*( ) -</sup> Number of pieces in total length.
\*\*Fiber length was too short to evaluate.
\*\*\*From near-field photomicrographs.

Table 2.3.3-1. Precision Large Core Multimode Fiber Data (continued).

## Preform Failures

**BG 21435** 

EG 21452

EG 21355

EG 21506

EG 21517

The optical results for these fibers were very encouraging in terms of their potential use in couplers and systems. However, the fabrication yield was low due to poor quality substrate tube materials. If large volume applications develop for these fibers, then studies of alternative materials could reduce fiber cost through improved yield.

The optical results for the low NA single-mode fibers are listed in Table 2.3.3-2. This chronological listing shows fiber NA, od, and attenuation at three wavelengths. It should be noted that the attenuation of several of the fibers was very high, especially at longer wavelengths. Internally funded studies conducted very recently indicate that the second mode cut-off wavelength  $(\lambda_{\rm CO})$  calculated from the NA and core diameter is about 25% higher than the  $\lambda_{\rm CO}$  actually measured using an attenuation technique. This may explain the high losses noted for many of these fibers since the light is propagating in a low V or in a highly radiative condition. In order to transmit in an optimum condition, the low NA fibers should be redesigned to have a core diameter measured as about 5.5  $\mu m$  to give a  $\lambda_{\rm CO}$  equal to 0.57  $\mu m$  corresponding to 90% of  $\sqrt{\rm co}$  at 0.63  $\mu m$ .

Table 2.3.3-2. Fiber Optical Data Summary: 0.10 NA Single-Mode Fibers.

Preform No	NA*	Fiber od (um)	Lg (B)	Attenuat 0.63 µm	ion (±0.5 0.85 μm	dB/km) 1.03 μm
BMT-21153A	0.119	82	104	8.0	32.3	NT**
EMT-21153B	0.119	80	137	15.7	14.9	nt
EMT-21174	0.117	76	120	10.7	8.2	105
EMT-21178	0.112	81	118	6.9	11.6	NT
EMT-21182	0.113	81	118	8.3	6.6	nt
EMT-21186	0.104	85	114	13.9	nt	nt
EMT-21227A	0,110	80.5	120	49.1	nt	nt
EMT-21227B	-	80	560	20.8	NT	nt
EMT-21229	0.112	80.5	120	21.41	nt	nt
EMT-21254	0.115	79.5	512	NE ***	ne	NE

<sup>\*</sup>NA = Numerical aperture.

\*\*NT = No measurable transmission.

<sup>\*\*\*</sup>NE = Not evaluated.

The progress in development of the precision high NA single-mode fibers is shown in Table 2.3.3-3. Achievement of the goals for core diameter of 2.2  $\mu m$  and NA of 0.20 in an 80  $\mu m$  od fiber was previously shown. However, all these fibers were multimode at 0.63  $\mu m$ . The  $\lambda_{CO}$  measurement was made on several of these fibers (as shown in Table 2.3.3-4) and was found to be consistent at about 0.70  $\mu m$ . As a result, additional preforms (21353-21345) were fabricated with the same NA but a correspondingly smaller core. The  $\lambda_{CO}$  was successfully adjusted to near the desired value of 0.57  $\mu m$  so that single-mode operation at 0.63  $\mu m$  resulted.

Table 2.3.3-3. Piber Optical Data Summary: 0.20 NA Single-Mode Pibers.

Preform No	NA*	Fiber od (um)	Lq (m)	Attenuation (±0.5 dB/km) 0.63 μm 0.85 μm 1.03 μm
BHT-21165	0.10	-		
EMT-21167	0.11	-	-	
EMT-21176A	0.13	87	234	18.1 7.2 NE
EMT-21180A	0.19	107	114	Multimode at 0.63 $\mu$ m
BMT-21180B	0.19	103	102	Multimode at 0.63 $\mu m$
EMT-21180C	0.19	87	750	Multimode at 0.63 $\mu m$
EMT-21183A	0.19	83	117	9.4 3.2 2.6
EMT-211838	-	82	1125	NT 7.7 4.7
EMT-21187A	0.19	86	120	Multimode at 0.63 $\mu$ m
BMT-21187B	0.19	85	110	50.3 7.75 4.96
EMT-21187C	0.19	86	1535	Multimode at 0.63 $\mu m$
BMT-21228	0.17	80	2280	NE
EMT-21231	0.20	79	1580	Multimode at 0.63 µm
EMT-21241	0.20	80	1300	Multimode at 0.63 $\mu m$
BMT-21241A	-	-	595	NT 19.1 17.2
BMT-21257	0.20	81	1365	Multimode at 0.63 µm
BMT-21353	0.19	80 ±0.6	2155	NT
<b>ЕМН-21368</b>	0.21	80 ±1.0	1910	Low signal 17.02 13.8
RMH-21386	0.19	80 ±1.0	2600	52.89 14.91 9.20
EMR-21345	0.20	80 ±1.2	2970	85.46 21.50 12.63

<sup>\*</sup>Mumerical aperture.

**E** 

Table 2.3.3-4. Second Mode Cut-Off Wavelengths for Several High WA Single-Mode Pibers.

Preform No	Cut-Off Wavelength (µm)
BHT-21241	0.6930
ENT-21231A	0.7040
BMT-21231B	0.6980
BMT-21257A	0.7040

## 3.0 CONCLUSIONS AND RECOMMENDATIONS

The experimental efforts performed during this contract have clearly established that both high and low NA single-mode fibers and large core multimode fibers can be fabricated with a degree of precision which approaches practical measurement limitations. Specific conclusions have been compiled below:

- a. Fabrication of precision large core 100  $\mu m$  ±1  $\mu m$  graded-index fibers is feasible. The resulting fibers will be within the required ±1% od and core tolerance allowed.
- b. Fibers made with precision Vycor <sup>®</sup> tubing are fabricated at low yields because of bubbles developing in the preforms.
- c. Dimensional tolerances of better than ±1% on high NA and low NA single-mode fibers can be met.
- d. Of all the procedures evaluated for checking the od of the fiber, the laser micrometer was considered to be most accurate.
- e. Precision bore and od substrate tubing and fabrication processes where strict mass flow control and traverse linearity are maintained are necessary conditions for the fabrication of precision preforms.
- f. Precision preforms, a uniform heat source, and rapid diameter sensing are necessary conditions for the drawing of precision fibers.

Based on the results achieved and observations made during this contract, EOPD offers the following recommendations to further

improve the performance of precision fibers.

- a. Investigate the causes of bubbles in precision Vycor wased in large core fiber fabrication. This inquiry would also involve the evaluation of other types of Vycor or alternate borosilicate glasses.
- b. Improve the optical performance of both the low and high NA single-mode fibers by adjusting the measured  $\lambda$  to give 90% /co of the operating wavelength desired. This could be at either 0.63 µm, 0.83 µm, or both.
- c. Begin work on single-mode fibers for operation at 1.2 or 1.3  $\mu m$  in anticipation of future long length, high sensitivity sensor applications. Approaches to this fiber involve decreased amounts of boron and/or phosphorous and can utilize fluorine as a dopant to reduce attenuation at the longer wavelengths.

